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पैट्रोलियम और उसके उत्पादों की परीक्षण पद्धति

[पी : 60]

कोण भेदनमापी द्वारा स्नेहक ग्रीसों की गाढ़ता

(दूसरा पुनरीक्षण)

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

[P : 60]

CONSISTENCY OF LUBRICATING GREASES BY CONE PENETROMETER

(Second Revision)

First Reprint MAY 1999

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December 1994

Price Group 4

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard [P : 60] (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This method of test was first published in 1967 based on the joint publication ASTM D 217-60T and IP 50/64 T which was subsequently revised in 1971. In the first revision procedure for the determination of undisturbed penetration had been included and the precision limits were modified to align the method with the joint publication ASTM D 217-68 and IP 50/69.

This revision has been taken up as a result of the review of the method of test with the joint publication of ASTM D 217-88 and IP 50/88. In this revision the undisturbed penetration method has not been given separately instead has been included as an Annex to this method of test mainly because of the fact that the use of undisturbed penetration as a quality control test does not involve the transfer of grease samples from one container to another prior to testing and the undisturbed penetration on grease samples can be measured using the general procedure. Precision limits have also been modified to align this method with the joint publication ASTM D 217-88 and IP 50/88.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

[P : 60]

CONSISTENCY OF LUBRICATING GREASES BY CONE PENETROMETER

(Second Revision)

1 SCOPE

1.1 These test methods cover four procedures for measuring the consistency of lubricating greases by penetration of the standard cone. These test methods include procedures for the measurement of unworked, worked, prolonged worked, and block penetrations. Penetrations up to 475 may be measured. Undisturbed penetrations are described in the Annex B.

NOTES

1 The National Lubricating Grease Institute classified greases according to their consistency as measured by the worked penetration. The classification is as follows:

LGI Consistency Number	Worked Penetration Range, 25°C
000	444 to 475
00	400 to 430
0	355 to 385
1	310 to 340
2	265 to 295
3	220 to 250
4	175 to 205
5	130 to 160
6	85 to 115

2 The procedure for the measurement of penetration of petrolatum is described in IS 1448 [P : 92] : 1979. 'Methods of test for petroleum and its products : Part 92 cone penetration of petrolatum

1.2 Unworked penetration do not generally represent the consistency of greases in use as effectively as do worked penetrations. The latter are usually preferred for inspecting lubricating greases.

1.3 Penetrations of block greases can be obtained on those products which are sufficiently hard to hold their shape. These greases generally have penetrations below 85.

1.4 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 DESCRIPTION OF TERMS SPECIFIC TO THIS STANDARD

2.1 Penetration of Lubricating Grease

The depth, in tenths of a millimeter, that the standard cone penetrates the sample under prescribed conditions of weight, time, and temperature.

2.2 Working

The subjection of a lubricating grease to the shearing action of the standard grease worker.

2.3 Unworked Penetration

The penetration at 25°C of a sample of lubricating grease that has received only minimum disturbance in transferring to a grease worker cup or dimensionally equivalent rigid container.

NOTE — The penetrations of soft grease are dependent upon the diameter of the container. Therefore, greases having unworked penetrations above 265 should be tested in containers having the same diameter limitations as those of the worker cup. The results on greases having penetrations below 265 are not significantly affected if the diameter of the container exceeds that of the worker cup.

2.4 Worked Penetration

The penetration of a sample of lubricating grease that has been brought to 25°C subjected to 60 double strokes in a standard grease worker, and penetrated without delay.

2.5 Prolonged Worked Penetration

The penetration of a sample of lubricating grease after it has been worked more than 60 double strokes in a standard grease worker at a temperature of 15 to 30°C. After the prescribed number of double strokes the worked and contents are brought to 25°C, worked an additional 60 double strokes, and penetrated without delay.

2.6 Block Penetration

The penetration at 25°C of a sample of lubricating grease that is sufficiently hard to hold its shape, determined on the freshly prepared face of a cube cut from a block of the grease.

3 SUMMARY OF TEST METHODS

The penetration is determined at 25°C by releasing the cone assembly from the penetrometer and allowing the cone to drop freely into the grease for 5 s.

4 SIGNIFICANCE AND USE

Worked penetration results may be used to establish the consistency of lubricating greases within the NLGI consistency grades shown in Note under **1.1**. The change in worked penetration results of a grease after prolonged working are believed to be a measure of its shear stability, under the conditions of the test. All four test procedures are widely used for specification purposes, however no correlation with field service has been established.

APPARATUS

5.1 Penetrometer

An instrument similar to that shown in Fig. 1 designed to measure in tenths of a millimeter the depth to which the standard cone penetrates the grease. The cone assembly of the table of the penetrometer shall be adjustable to enable accurate placement of the tip of the cone on the level surface of the grease while maintaining "zero" reading on the indicator. When released, the cone should fall without appreciable friction. The tip of the cone should not hit the bottom of the sample container. The instrument shall be provided with levelling screws and a spirit level to maintain the cone shaft in a vertical position.

NOTE — Both the penetrometer shaft and the rack engaging the measuring dial should be of sufficient length to test greases with penetrations as high as 620. If only penetrations less than 400 are to be measured, the penetrometer may be designed such that when released, the cone falls for at least 40.0 mm.

5.2 Standard Cone

As specified in **A-1.1**. An optional cone, described in **A-1.2**, may be used for penetrations less than 400.

5.3 Grease Worker

As specified in **A-1.3**.

5.4 Mechanical Grease Worker

A machine designed to operate the plunger of the grease worker mechanically as specified in **A-1.3**. This apparatus is essential for the measurement of prolonged worked penetration.

5.5 Grease Cutter

As specified in **A-1.4**.

5.6 Water or Air Bath

Capable of regulation to 25 ± 0.5°C and designed to bring the assembled worker to test temperature conveniently. If a water bath is to be used for samples for unworked penetration, means should be provided for protecting the grease surface from water and for maintaining the air above the sample at test temperature. An air bath is preferred for bringing block greases to test temperature, but a tightly sealed container placed in a water bath will suffice. A constant-temperature test room may be used instead of the water or air bath.

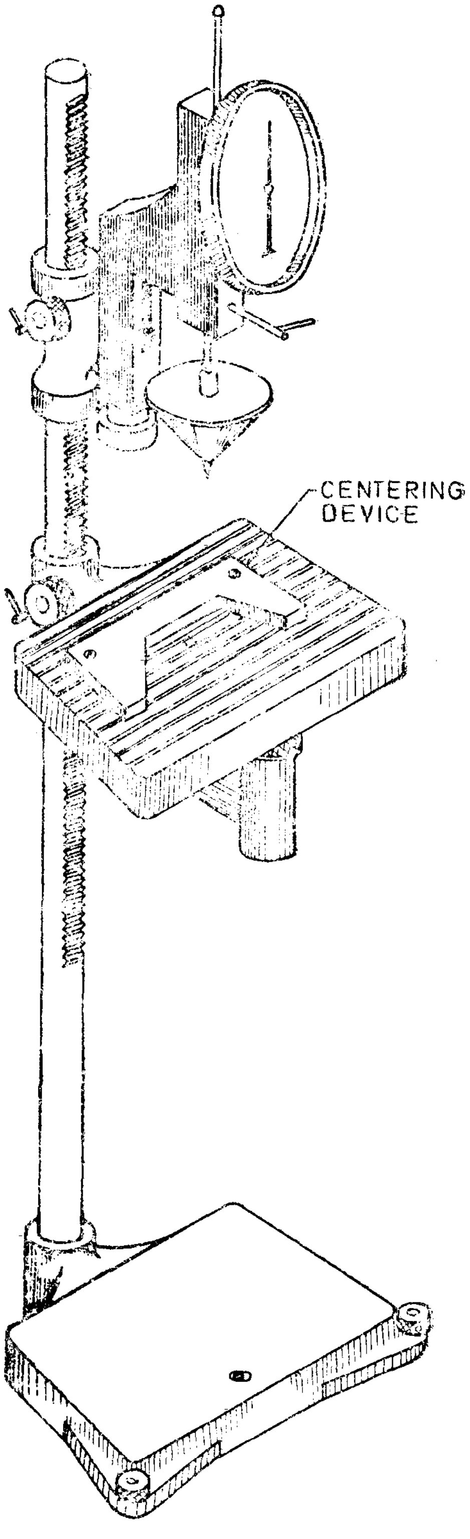


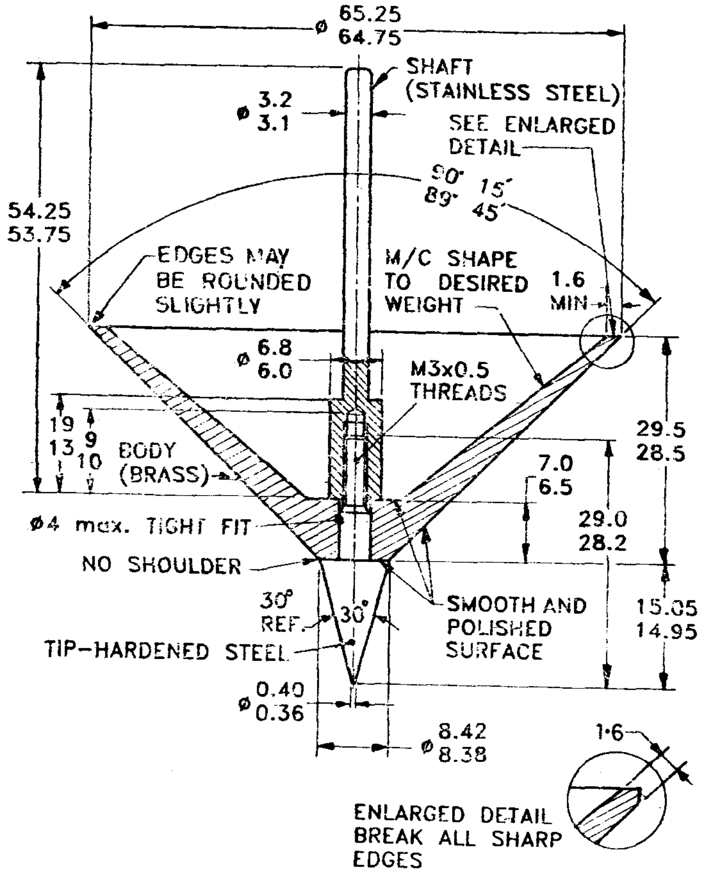
FIG. 1 PENETROMETER

5.7 Spatula

Corrosion-resistant, having a stiff blade 32 mm wide and at least 150 mm long.

5.8 Overflow Ring (Optional)

As described in **A-1.5**. Both the grease removed



All dimensions in millimetres.

FIG. 2A OPTIONAL PENETROMETER CONE

in scraping and the grease forced by the penetrometer cone to overflow may be returned to provide a full cup for the next test. The overflow ring is convenient for returning overflowed grease to the worker cup.

6 PROCEDURE FOR UNWORKED PENETRATION

6.1 Sample

Sufficient sample (at least 0·4 kg) to overfill the cup of the standard grease worker is required. If sample size is insufficient and penetration ranges from NLGI Grade 0 to 4, use Test 'Method IS 1448 [P : 116] : 1992 'Methods of test for petroleum and its products : Part 116 Determination of cone penetration of lubricating grease using one-quarter and one-half scale cone'. If the transferred penetration is above 200, at least three times the amount needed to fill the cup is required.

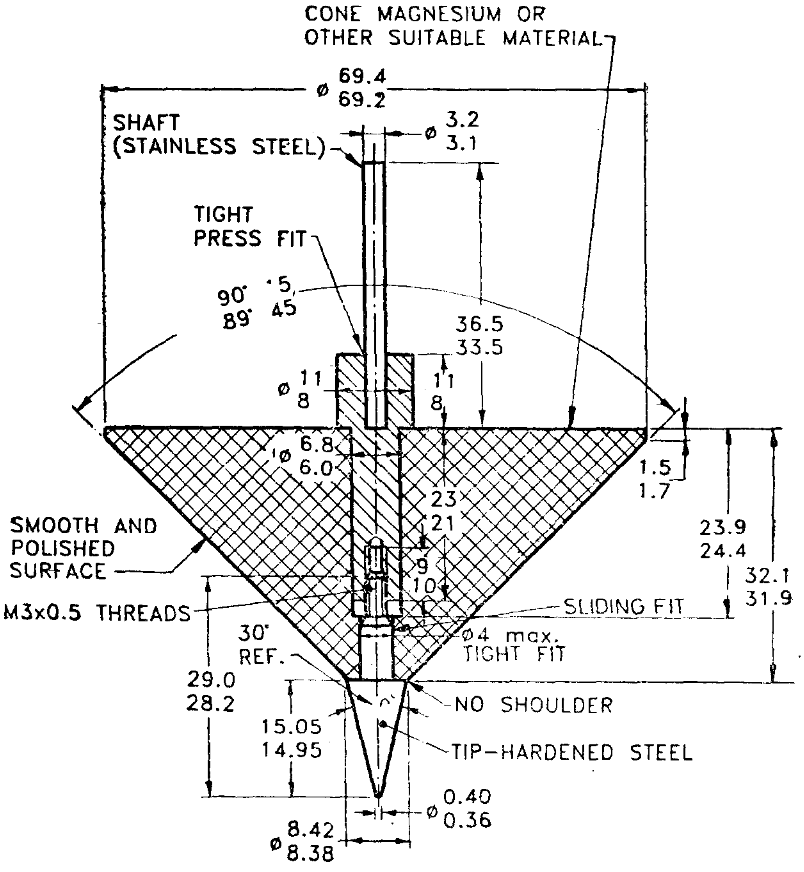
6.2 Preparing Sample for Measurement

Place the empty grease worker cup and an appropriate amount of the sample in a metal container in the water or air bath maintained at 25°C for sufficient time to bring the temperature of the sample and the worker cup to 25 ± 0·5°C. Transfer the specimen, preferably in one lump, to overfill the cup of the grease worker (*see* Note under **2.3**). Make this transfer in such a manner that the grease will be worked as little as possible. Jar the cup to drive out trapped air and pack the grease with the spatula, with as little manipulation as possible to obtain a cupful without air pockets. Scrape off the excess grease extending above the rim by moving the blade of the spatula, held inclined toward the direction of motion at an angle of 45°, across the rim of the cup. Do not perform any further levelling or smoothing of the surface throughout the determination of unworked penetration and determine the measurement immediately.

NOTE — If the initial sample temperature differs from 25°C by more than about 8°C, or if an alternative method of adjusting the sample to 25°C is used, allow sufficient additional time to assure that the specimens is at 25 ± 0·5°C before proceeding. Also if the sample is larger than 0·4 kg, allow sufficient additional time to assure that the specimen is at 25 ± 0·5°C. Testing may proceed if the specimen is at a uniform temperature of 25 ± 0·5°C.

6.3 Cleaning Cone and Shaft

Clean the penetrometer cone carefully before each test. Bending of the cone shaft can be avoided by holding it securely in its raised position while cleaning. Do not permit grease or oil on the penetrometer shaft, as they can cause drag on the



NOTE — The total weight of the cone shall be 102·5±0·05 gm and total weight of its movable attachments shall be 47·5±0·05 gm.

All dimensions in millimetres.

FIG. 2B STANDARD PENETROMETER CONE

shaft assembly. Do not rotate the cone, as this may wear on the release mechanism.

6.4 Penetration Measurement

Place the cup on the penetrometer table, making certain that it cannot teeter. Set the mechanism to hold the cone in the "zero" position, and adjust the apparatus carefully so that the tip of the cone just touches the surface at the centre of the test specimen. Watching the shadow of the cone tip is an aid to accurate setting. For greases with penetrations over 400, the cup (Fig. 3) must be centred to within 0·3 mm of the tip of the cone. One way to centre the cup accurately is to use a centering device (Fig. 1). Release the cone shaft rapidly, and allow it to drop for 5·0 ± 0·1 s. The release mechanism should not drag on the shaft. Gently depress the indicator shaft until stopped by the cone shaft and read the penetration from the indicator.

6.4.1 If the sample has a penetration over 200, centre the cone carefully in the cup; this sample can then be used for only one test. If the sample has a penetration of 200 or less, perform three tests in a single cup, spacing these tests on three radii 120° apart and midway between the centre and side of the cup so that the cone will neither strike the side of the container nor impinge on the disturbed area made in a previous test.

6.5 Additional Testing

Make a total of three tests on the specimen (either in three cups or in one, as described in 6.4), and report the average value, to the nearest unit, as the unworked penetration of the specimen.

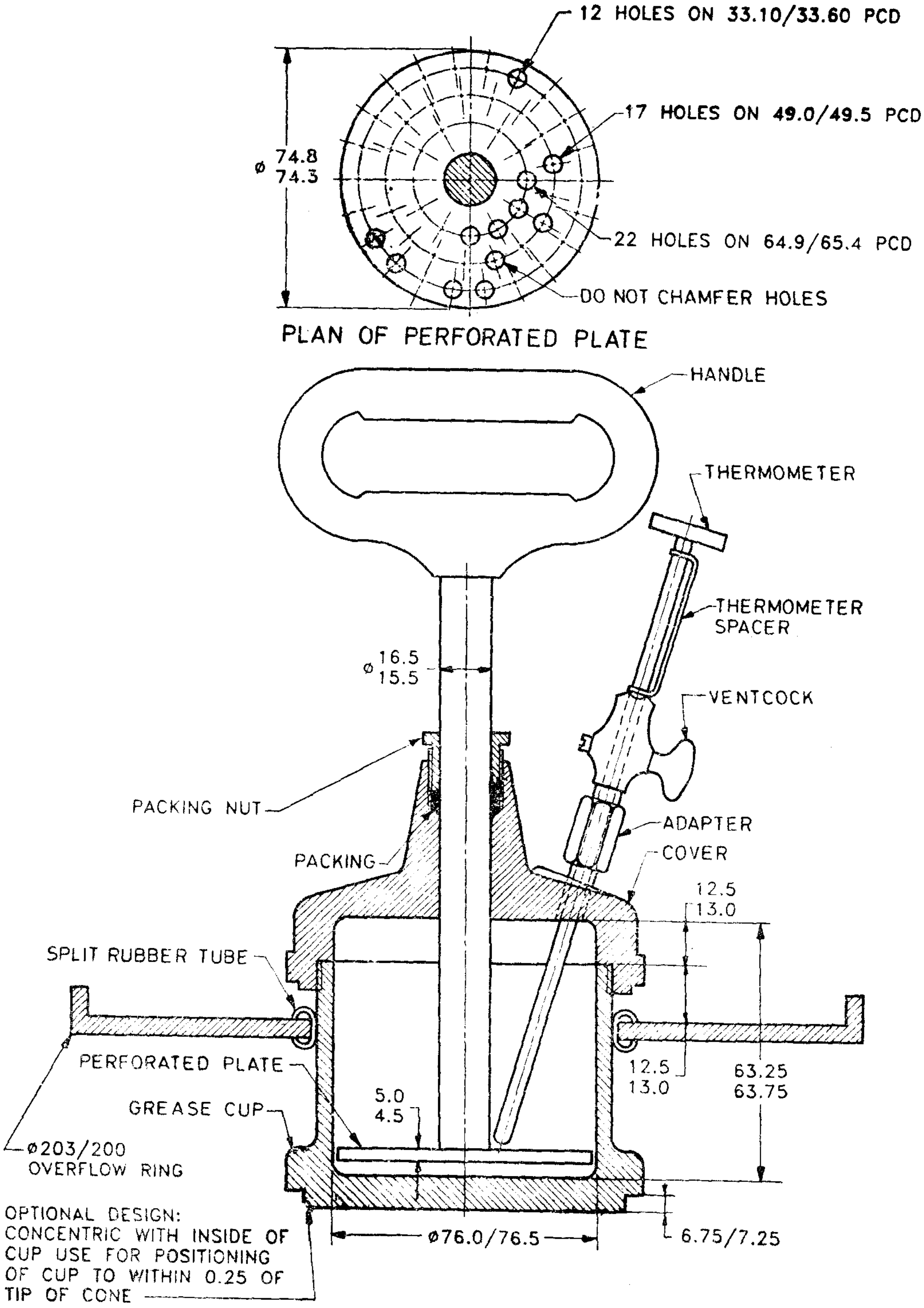
7 PROCEDURE FOR WORKED PENETRATION

7.1 Sample

Sufficient sample (at least 0·4 kg) to overfill the cup of the standard grease worker is required. If sample size is insufficient and penetration ranges from NLGI Grade 0 to 4, use Test Method IS 1448 [P : 116] : 1992.

7.2 Working

Transfer sufficient specimen to the cup of the clean grease worker to fill it heaping full



All dimensions in millimetres.

FIG. 3 GREASE WORKER

(mounded up about 13 mm at the centre), avoiding the inclusion of air by packing with the spatula. Jar the cup from time to time as it is being packed to remove any air inadvertently entrapped. Assemble the worker and, with the vent cock open, depress the plunger to the bottom. Insert a thermometer through the vent cock so that its tip is in the centre of the grease. Please the assembled worker in the water or air bath maintained at 25°C until the temperature of the worker and contents are 25 ± 0·5°C as indicated by the thermometer. Then remove the worker from the bath and wipe off any excess water adhering to its surfaces. Remove the thermometer and close the vent cock. Subject the grease to 60 full (67 to 71·5 mm) double strokes of the plunger, completed in about 1 minute, and return the plunger to its top position. Open the vent cock, remove the top and plunger, and return to the cup as much of the grease clinging to the plunger as may readily be removed. As the worked penetration of a lubricating grease may change significantly on standing, proceed in accordance with **7.3**, **7.4** and **7.5** immediately.

NOTE — If it is desired to immerse the position of the worker above its closure, take care that the lid is watertight in order to prevent the entrance of water to the worker.

7.3 Preparing Specimen for Measurement

Prepare the worked sample in the cup for testing so that a uniform and reproducible structure of the grease will be obtained. Jar the cup sharply on the bench or floor and pack the grease down with a spatula to fill the holes left by the plunger and to remove any air pockets (Note 1). Scrape off the excess grease extending above the rim of the cup by moving the blade of the spatula, held inclined toward the direction of motion at an angle of 45°, across the rim of the cup, retaining the portion removed (Note 2).

NOTES

1 The jarring should be as vigorous as required to remove the entrapped air without splashing the specimen from the cup. In performing these operations, a minimum of manipulation should be used, as any agitation of the grease may have the effect of increasing the working beyond the specified 60 strokes.

2 Particularly when testing soft greases, retain the grease removed from the cup in scraping to provide a full cup for subsequent tests. Keep the outside of the rim of the cup clean so that the grease forced by the penetrometer cone to overflow the cup may be returned to the cup prior to preparing the specimen for the next test.

7.4 Penetration Measurement

Determine the penetration of the specimen as described in **6.3** and **6.4**.

7.5 Additional Tests

Immediately make two more tests in succession on the same specimen. Return to the cup the portion previously removed with the spatula, then repeat the operations described in **7.3** and **7.4**. Report the average of the three tests, to the nearest 0·1 mm, as the worked penetration of the specimen.

8 PROCEDURE FOR PROLONGED WORKED PENETRATION

8.1 Temperature

Maintain the temperature of the room used for the test within the range from 15 to 30°C. No further control of the worker temperature is necessary; but before starting the test, the grease should have been in the room for sufficient time to bring its temperature within the range from 15 to 30°C.

8.2 Working

Fill a clean grease-worker cup and assemble the worker as described in **7.1**. Subject the grease specimen to the prescribed number of double strokes.

NOTE — In order to minimize leakage during working, special attention must be paid to the gland in the worker cover.

8.3 Penetration Measurement

Immediately after the working is concluded, place the worker in the constant temperature water or air bath to bring the test specimen temperature to 25 ± 0·5°C within 1·5 h. Remove the worker from the bath and subject the grease to a further 60 double strokes. Prepare and penetrate the samples as described in **7.4** and **7.5**, except for reporting the average of three tests as the prolonged worked penetration for the prescribed number of double strokes.

9 PROCEDURE FOR BLOCK PENETRATION

9.1 Sample

Obtain a sufficient size sample of the grease, which must be hard enough to hold its shape, to permit cutting from it a 50-mm cube as a test specimen.

9.2 Preparing Sample for Measurement

By means of the specified grease cutter, cut as a test specimen from the sample at room temperature a cube about 50-mm on the edge. While holding this specimen so that the unbeveled edge of the cutter is toward it, slice off a layer about 1·5 mm in thickness from each of the three faces adjacent to a single corner, which may be truncated for identification. Take care not to touch those portions of the newly exposed faces which are to be used for testing or to set a prepared face against the base plate or guide of the cutter. Bring the temperature of the finished specimen to 25 ± 0·5°C by placing it in a constant temperature air bath maintained at 25°C for at least 1 h. (*See* Note under **6.2**).

NOTE — The testing of three faces is intended to equalize in the final value the effect of fibre orientation in testing fibrous greases. Smooth-textured, non-fibrous greases may be tested on one face only, when agreed upon between interested parties.

9.3 Penetration Measurement

Place the test specimen on the penetrometer table with one of the prepared faces upward, and press it down by the corners to make it rest level and firmly on the table so that it cannot teeter during the test. Set the mechanism to hold the cone in 'zero' position, and adjust the apparatus carefully so that the tip of the cone just touches the surface at the centre of the test sample. Determine the penetration as described in **6.3** and **6.4**. Make a total of three tests on the exposed face of the specimen, locating the tests at least 6 mm from the edge and as far apart as possible without impinging on any touched portion, air hole, or other apparent flaw in the surface. If the result of these tests differs from the others by more than three units, make additional tests until three values agreeing within three units are obtained. Average these three values for the face being tested.

9.4 Additional Tests

Repeat the procedure described in **9.3** on each of the other prepared faces of the specimen. Report one third of the sum of the averages for the three faces, to the nearest unit, as the block penetration of the grease under test.

10 PRECISION

10.1 Repeatability

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values in Table 1 in only one case in twenty.

10.2 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values in Table 1 in only one case in twenty.

Table 1 Repeatability and Reproducibility

Penetration	Peneatration Range	Repeatability One Operator and Apparatus	Reproducibility Different Operators and Apparatus
Unworked	85 to 475	8 units	19 units
Worked	130 to 475	7 units	20 units
Prolonged worked	130 to 475	15 units*	27 units*
Block	under 85	7 units	11 units

*Determined at 100 000 double strokes within 15 to 30°C ambient temperature range.

ANNEX A

(*Clause* 5)

(MANDATORY INFORMATION)

A-1 APPARATUS

A-1.1 Standard Cone, for Measuring Penetrations Up to 475

Consisting of a conical body of magnesium or other suitable material with detachable, hardened steel tip, shall be constructed to conform to the tolerances shown in Fig. 2B. The total weight of the cone shall be 102·5 ± 0·05 g and that of its movable attachments shall be 47·5 ± 0·05 g; the attachments consist of a rigid shaft having a 'stop' at its upper end and suitable means, at its lower end, for engaging the cone. The interior construction may be modified to achieve the specified weight, provided that the general contour and weight distribution are not altered. Polish the outer surface to a very smooth finish.

A-1.2 Optional Cone, for Measuring Penetration Up to 400

Consisting of a conical body of brass or corrosion-resistant steel with detachable, hardened steel tip, shall be constructed to conform to the tolerances shown in Fig. 2A. The total weight of the cone shall be 102·5 ± 0·05 g and that of its movable attachments shall be 47·5 ± 0·05 g; the attachments consist of a rigid shaft having a 'stop' at its upper end and suitable means, at its lower end, for engaging the cone. The interior construction may be modified to achieve the specified weight, provided that the general contour and weight distribution are not altered. Polish the outer surface to a very smooth finish.

A-1.3 Grease Worker

Conforming to the dimensions given in Fig. 3. The dimensions not shown may be altered and other methods for fastening the lid and securing the worker may be used. The worker may be constructed for either manual or mechanical operation. The design must be such that a rate of 60 ±5 strokes per minute with a minimum length of 67 mm, can be maintained. A suitable temperature indicator, standardized at 25°C, shall be provided for insertion through a vent cock. If mechanically operated, the grease worker must be provided with a presenting counter to permit the apparatus to be automatically stopped after any required number of double strokes up to 999 99.

A-1.4 Grease Cutter

Having a sharp, rigidly mounted, beveled blade. It is necessary that the blade be straight and sharpened.

A-1.5 Overflow Ring

Is a useful aid for returning displaced grease to the worker cup. The overflow ring shall be positioned at least 13 mm below the rim of the cup while making a penetration measurement. A rim of 13 mm high is helpful.

ANNEX B

(*Clause* 1.1)

(NONMANDATORY INFORMATION)

UNDISTURBED PENETRATION

B-1 Undisturbed penetration is defined as the penetration at 25°C of a sample of grease in its container with no disturbance. Typical uses of the undisturbed penetration are : (1) to control the consistency of certain greases during manufacture and (2) to assess the degree to which a grease develops a 'false body' or 'set' upon prolonged storage. The use of undisturbed penetration as a quality control test does not involve the transfer of grease samples from one container to another prior to testing. However, in evaluating the effect of prolonged storage upon a lubricating grease, samples may be repacked prior to storage in an appropriately sized container. Parties interested in measuring the undisturbed penetration on grease samples should use the general procedure described in **6.3**, **6.4** and **6.5** and establish mutually agreeable conditions for storage and container size.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

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